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BUREAU OF STANDARDS

S. W. STRATTON, DIRECTOR

No. 41

LEAD ACETATE TEST FOR HYDROGEN SULPHIDE IN GAS

BY

R. S. McBRIDE, Associate Chemist

and

J. D. EDWARDS, Assistant Chemist

Bureau of Standards

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LEAD ACETATE TEST FOR HYDROGEN SULPHIDE IN GAS

By R. S. McBride and J. D. Edwards

CONTENTS

I. PURPOSE OF INVESTIGATION

The regulations now in force in 10 States and in 27 large cities in other States regarding the purity of manufactured gas prescribe that the gas must be free from hydrogen sulphide or must contain not more than a "trace" of that substance. For the intelligent enforcement of such regulations definite knowledge regarding the significance of the various methods of testing for hydrogen sulphide in gas is necessary.

Many tests for hydrogen sulphide, both qualitative and quantitative, have been proposed. The test with lead acetate paper, however, has been almost universally used in gas testing, and it seems to be the one best adapted for that purpose. At the present time there is a wide variation in the method of carrying out this test. This lack of uniformity, together with the absence of definite information which would enable one to compare results of tests made in different ways, has given rise to misunderstanding and dissatisfaction. Some of the tests are designed to detect as small traces of hydrogen sulphide as possible, while others aim to give a negative test when a permissible amount is not exceeded. It is, therefore, necessary to understand the purpose of a given test before forming an opinion as to its merits.

The purpose of this investigation has been to study the effect of all of the important variables which may affect the results of different tests, in order to ascertain the relative sensitiveness of the methods and the minimum amount of hydrogen sulphide which can be detected by certain of these. It has also been our purpose to fix upon a definite test which should possess the following characteristics: It should be of suitable sensitiveness; it should be exactly reproducible; the apparatus necessary should be simple and inexpensive; and the method should be rapid and easy of application. It is desirable that such a test, although qualitative in principle, should be of quantitative significance.

II. PREVIOUS WORK

Only three papers have been found which bear directly upon the problem; these are by W. J. Dibdin and R. G. Grimwood,[1] R. Forbes Carpenter,[2] and Charles J. Ramsburg.[3] In the discussion of the first of these three before the English Society of Public Analysts the fact was brought out by Hehner that some additional work had been done by Dr. Rideal and by himself. However, so far as we have been able to determine, there are no published reports of this work. The methods employed, the results obtained, and the conclusions drawn in the three articles will be briefly summarized. The significance of this earlier work will be discussed in a later section of the present paper, on the quantitative significance of the various test methods.

1. DIBDIN AND GRIMWOOD

Dibdin and Grimwood employed for the preparation of their gas samples a 15-liter bottle, into which measured quantities of hydrogen sulphide were introduced and allowed to diffuse through the gas during a period of 30 to 45 minutes. Then, by inflating an india-rubber balloon, which was suspended from the rubber stopper in the neck of the bottle by means of a small glass tube, the gas was forced out of the bottle into the apparatus, where it impinged upon the lead acetate paper. When tests made on gas, taken from the top and bottom of the bottle, showed the same coloration, the mixing of the hydrogen sulphide with the gas was considered to be complete. When one series of tests had been completed, a gas of lower hydrogen sulphide content was made. A measured volume of the previous mixture was withdrawn and the residue washed out by passing coal gas through the reservoir until it gave no test for hydrogen sulphide. The measured volume of contaminated gas was then returned to the bottle, diffusion allowed to take place, and the gas reexamined. Successive dilutions were made to produce a gas mixture containing 1 volume of hydrogen sulphide in 10 000 000 000 volumes of gas.

[1] Analyst, **27**, p. 219; 1902. Reprinted in J. of Gas Lighting, **80**, p. 687; 1902.

[2] Forty-fourth Ann. Report on Alkali, etc., Works by the Chief Inspector, p. 81; 1907. Reprinted in J. of Gas Lighting, **103**, p. 167; 1908.

[3] Proc. Amer. Gas Inst., **4**, p. 453; 1909.

The test papers were dipped in a 12 per cent solution of lead acetate, held for a few minutes, while still wet, in an atmosphere of ammonia, then dried, and used in the dry condition. Table 1 shows the time required to produce a coloration when the gas impinged upon the paper at the rate of 0.5 cubic foot per hour.

TABLE 1

Tests Reported by Dibdin and Grimwood[4]

Grains of H_2S per 100 cubic feet of gas[5]	Volumes of mixture per 1 volume of H_2S	Time to give stain at different distances of paper from nozzle (minutes and seconds)			
		Close to—	$\frac{1}{8}$ inch	$\frac{1}{4}$ inch	$\frac{1}{2}$ inch
63	1000	At once	At once	At once	At once
6.3	10 000	0– 2	0– 4	0– 8	0–12
.25	250 000	0– 5	0–10	0–15	0–18
.063	1 000 000	0– 8	0–15	0–30	....
.006	10 000 000	1– 7	1–30	1–50	2–20
.0006	100 000 000	5– 0	....	12– 0	....
.00006	1 000 000 000	11–30	....	21–30	....
.000006	10 000 000 000	>30	>30	>30	>30

[4] Loc. cit.

[5] The values given in this column have been calculated on the assumption that the original gas measurements were made at 60° F and 30″ barometric pressure, in order to give an approximate idea of Dibdin and Grimwood's results in grains per 100 cubic feet.

In the discussion of this paper before the Society of Public Analysts objections were raised to the use of the rubber balloon, because of the difficulty of freeing the rubber from hydrogen sulphide, as reported by Dr. Rideal, who had used goldbeater's skin in its place. Mr. Hehner stated that it was his experience that the goldbeater's skin also rapidly removed traces of hydrogen sulphide. It was also stated that Dr. Rideal was able to detect 1 volume of hydrogen sulphide in 5 000 000 volumes of gas, passing the gas at 5 cubic feet per hour through the official Referees apparatus of the old form, in which the gas impinged upon the test paper. The time of exposure is not stated; but since the capacity of his gas reservoir was only 40 liters, he was limited to an exposure of about 15 minutes.

2. CARPENTER

The work reported in the Forty-fourth Annual Report of the Alkali Works Inspector was undertaken, in order to determine the reliability of the test for detecting hydrogen sulphide in the gases from chimneys and flues of chemical works. The method of preparing the gas mixture was, in general, the same as that employed by Dibdin and Grimwood; but in place of the rubber balloon, one made of gold-beaters' skin was employed, and air, instead of gas, was used for diluting the hydrogen sulphide.

The following samples of paper were prepared and tested:

1. *"Dry" paper.*—Schleicher and Schüll filter paper (No. 589, black band), soaked in a 12 per cent solution of lead acetate and dried at 80 to 90° C.

2. *"Moist" paper.*—Filter paper of the same quality, soaked in 12 per cent lead acetate containing 5 per cent of glycerin and partially dried. Paper damp to feel.

3. *"Moist NH_3" paper.*—Sample "moist" paper (2) held before use in fumes of strong ammonia.

4. *"Dry paper wetted with NH_3."*—Sample "dry" paper wetted, before use on lower half of strip, with concentrated solution of ammonia.

The test papers were held "either suspended by platinum wire or doubled to receive the gas centrally" in the exit tube, which "was expanded above into a cylindrical funnel with stopcock 3 inches long in the body and 1¼ inches diameter * * * A porcelain Gooch crucible served as a loose-fitting cap to the funnel and carried the wire that held the test paper." The procedure and results are described as follows:

In each test 1100 cc of gas were used, the time being five minutes, equal to 13 200 cc per hour (nearly half a cubic foot). The moment when a discoloration was first noticeable on the paper was recorded, as well as the final effect. The more important results obtained are summarized as follows:

TABLE 2

Tests Reported by Alkali Works Inspector[6]

Test paper	Volumes of mixture per 1 volume H_2S	Color noticeable in seconds	Color after 5 minutes with 1100 cc of gas	Smell
1. "Dry." Gas impinges centrally	100 000	At once	Black	Decided
2. Ditto. Strip suspended	100 000	30	Deep brown	Do.
3. "Moist." Strip suspended	100 000	30	Deep brown, rather lighter than No. 2	Do.
4. "Dry." Suspended	1 000 000		Faint brown	Slight
5. "Moist, NH_3." Ditto	1 000 000		Slight brown, deeper than No. 4	Do.
6. Ditto. Impinges	1 000 000		Deep brown	Do.
7. "Dry." Impinges	10 000 000		Remains white	None
8. "Moist, NH_3." Ditto	10 000 000		Slight stain	Do.

[6] Loc. cit. Only tests made in absence of sulphur dioxide are included, since the others have little or no bearing upon the question under consideration. It may be noted, however, that: "Sulphur dioxide renders the lead paper test less sensitive to sulphuretted hydrogen. The effect commences with a concentration, SO_2, 0.5 grain per cubic foot (1 volume SO_2 per 2500 volumes), and increases with the proportion of sulphur dioxide present."

From a consideration of all the facts, of which the examples recorded above are typical, the following conclusions were drawn with respect to the results obtained by the described procedure:

1. *Condition of test paper.*—With gas up to 1 volume H_2S in 100 000 the condition of the test paper does not greatly affect the amount of discoloration. "Dry" paper, "moist" paper, and "moist NH_3" paper are about equally sensitive with suspended strip.

With gas 1 volume H_2S in 1 000 000 and upward, "moist NH_3" paper is most sensitive, and should alone be used.

2. *Direction of current.*—The test is far more sensitive when the gas is made to impinge directly on the surface of the paper. Thus, "moist NH_3" paper is only slightly discolored by air containing 1 volume H_2S in 1 000 000, when the flow is parallel to its surface; it is deeply stained when the gas impinges.

3. *Quantitative significance of results obtained with suspended strip.*—Stains of equal depth are obtained under the same conditions as regards quality of test paper, direction, and rate of flow of gaseous current, and proportion of sulphuretted hydrogen present.

Other conditions being equal, if the strength of gas be varied, the discoloration is greater as the proportion of sulphuretted hydrogen is increased, the difference of color obtained with gas 1 H_2S in 10 000, 20 000, 50 000, 100 000, etc., being easy to distinguish.

The limit of sensitiveness for "dry" paper is about 1 H_2S in 1 000 000; for "moist NH_3" paper, 1 H_2S in 10 000 000 with suspended strip.

3. RAMSBURG

The paper by C. J. Ramsburg reports on a number of tests, which were made to show the great difference in the intensity of color produced, when testing a given gas according to a number of different methods, as carried out in certain States and cities. It also contains a history of the subject of sulphur regulation in England, and gives the various official tests for total sulphur and hydrogen sulphide which have been used by the London gas Referees. The question of sulphur regulation, with particular reference to hydrogen sulphide, as followed in the United States, is discussed, and a number of State and municipal regulations on the subject are quoted.

The water in a 50 cubic foot gas holder was saturated with foul coal gas from which tar and ammonia had been removed, and by filling the holder with clean gas and allowing it to stand a gas was obtained which contained 0.53 grain of hydrogen sulphide per 100 cubic feet. The hydrogen sulphide was determined by absorption in an acid solution of cadmium chloride, the precipitated sulphide filtered off, oxidized to sulphate, and weighed as barium sulphate. Photographs are given of the test papers secured when testing this gas by the following six methods.

The test prescribed by the New York State commission, which was to hold a paper moistened with lead acetate in a current (or jet) of gas flowing at 5 cubic feet per hour for 30 seconds, showed a distinct coloration. Ramsburg evidently obtained this test with the gas impinging from a jet onto the paper, as may be seen from the form of stain shown in the photographic record of his results. The commission rule, quoted by him, provides for exposure to a "current of gas;" but the State law for second-class cities, not quoted, specifies exposure to a "jet of gas."

A stain of about the same depth as produced by the New York method was obtained with the "Connecticut test," which Ramsburg made "by allowing a stream of gas to flow through a round orifice not larger than $\frac{1}{32}$ of an inch in diameter, under a pressure not greater than 1.5 inches of water, and to impinge on a paper moistened with a solution of acetate of lead, placed at a distance of 1 inch from said orifice for a period of three minutes."

According to the "Boston method" a moist paper is held "in a small stream of gas ½ inch from the orifice for 60 seconds." A dark stain was produced when the test was carried out in this manner.

The present "London Gas Referees method," according to which a moist paper is suspended in the Referees apparatus (see Fig. 11) for three minutes, the gas flowing at the rate of 5 cubic feet per hour, showed no test in one minute; but a "rather distinct" test was obtained in three minutes, the color being, however, much lighter than in the three previous tests.

The "London Gas Referees method," used previous to 1906, specified the passage of 10 cubic feet of gas at the rate of 0.5 cubic foot per hour, the gas impinging on the paper. The test paper produced by this method was very dark.

The "Milwaukee method," which was to pass gas at the rate of 5 cubic feet per hour for one and one-half to two hours, through a piece of glass tubing 1 inch in diameter containing the test paper, also produced a deep black stain.

In the discussion of this paper before the Institute it was the opinion of many that the present tests were too sensitive. It seemed desirable to some to permit 1 grain of hydrogen sulphide per 100 cubic feet of gas; but such a specification would preclude the use of the simple tests, which are easy and quick of application. For this reason a qualitative method, the results of which would be roughly quantitative, was desired.

III. OUTLINE OF INVESTIGATION

From a consideration of the various methods used commercially for the detection of hydrogen sulphide and of the results obtained by the experimenters whose work is summarized above, as well as of preliminary tests made at the Bureau, the following variables were shown to be factors which must be considered in the investigation of these testing procedures:

1. The paper: Kind; method of its preparation, including the strength of lead acetate solution used, and the moisture content when used.

2. The gas: Its humidity, and the period and rate of flow.

3. The apparatus: Its form and size, as determining the circulation of gas with respect to the paper.

With gas mixtures containing various known amounts of hydrogen sulphide the several factors were studied one at a time; and results were obtained which indicate both the quantitative and the relative significance from an engineering or economic standpoint of the various methods of testing. It is hoped that this information will bring about a better understanding of the limitations which the various methods impose upon the gas maker. Having established the significance of the tests and the effect of the numerous variables, we have drawn up the specifications which are proposed for testing apparatus and methods.

IV. APPARATUS AND METHOD

1. PREPARATION OF GAS MIXTURES

One of the greatest difficulties met with in the study of the lead acetate test has been the preparation of a uniform gas mixture containing a known amount of hydrogen sulphide. None of the previous methods employed for this purpose, except that used by Ramsburg, has been free from very serious objections. For our work it was necessary to prepare 5 to 10 cubic feet of gas containing hydrogen sulphide in any desired amount. The following method of making such gas mixtures was devised:

The principle of operation is the mixing of known amounts of hydrogen sulphide, contained in some easily volatile solvent with known volumes of gas, this operation being conducted continuously and uniformly in the apparatus shown in Fig. 1. The gas first passes through a large tower A filled with sticks of potassium hydroxide, where any hydrogen sulphide originally present in the gas is removed. It then passes through the pressure regulator B, the wet meter C, and the glass tube F into the 30-liter mixing bottle H. The tower D, through which the gas could be

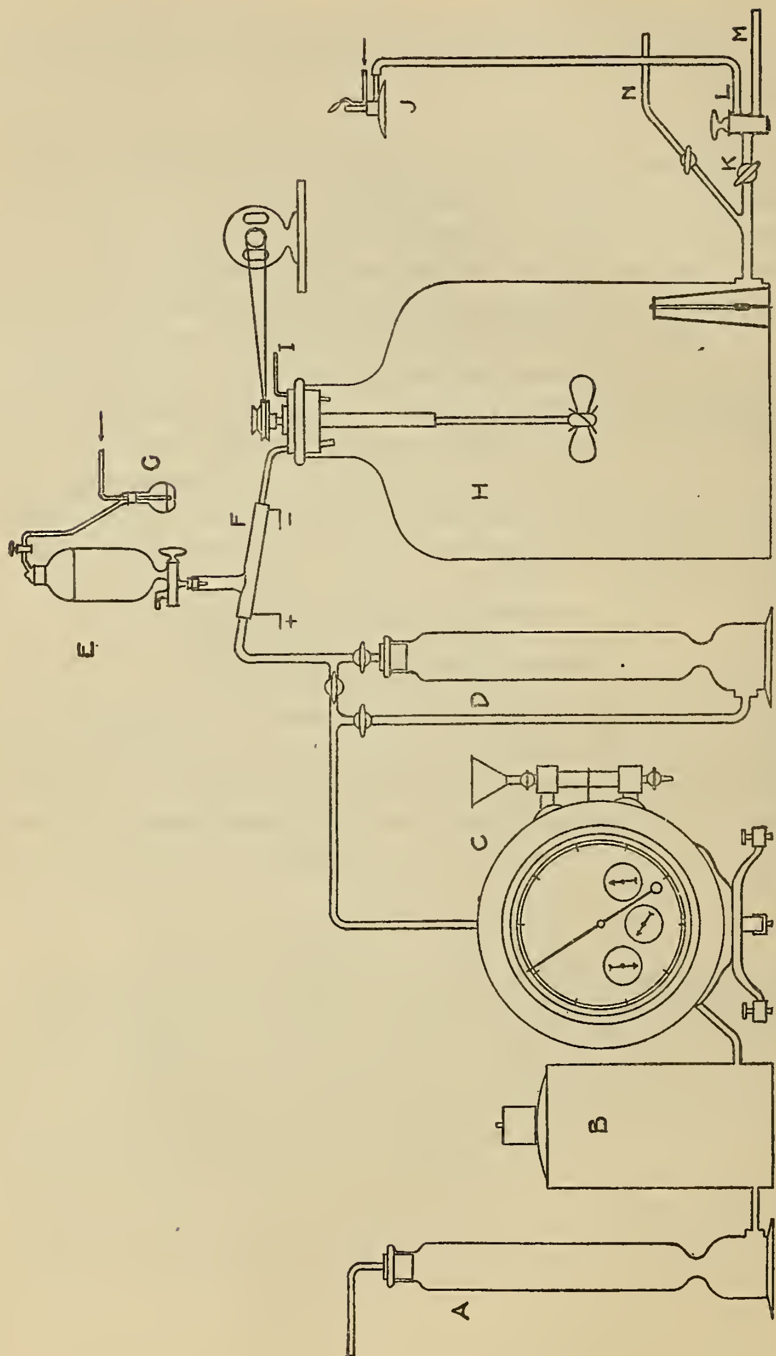

FIG. 1.—*Apparatus for preparation of gas mixtures.*

by-passed, served to dry the gas when desired. The solution of hydrogen sulphide in some solvent such as ether is held in the burette E, the tip of which is introduced through a rubber stopper into the side tube attached to F. The top of the burette is closed by a ground glass stopper to which is sealed a small stopcock. When a gas mixture is being prepared the solution is placed under slightly increased air pressure, and the stopcock at the bottom of the burette is adjusted so that the solution drips into the tube F at about 1 drop per second. The tube F, through which the gas passes at a uniform rate, is warmed by a small electric-heating coil, so that the hydrogen sulphide and its solvent are volatilized and carried along by the gas into the mixing bottle where a fan insures a thorough mixing. The increased pressure on the surface of the solution, necessary to make it drip regularly from the tip, is secured by bubbling air through a small wash bottle containing some of the same solution, the air being so regulated that one bubble enters the wash bottle for each drop of solution that leaves the burette. The pressure in the large mixing bottle is ascertained by a U gage attached to the small tube I, and the temperature is obtained from the thermometer suspended before the gas outlet at the bottom of the bottle.

After the gas has been thoroughly mixed in the large reservoir, it passes to a three-way cock L by means of which it may be diverted to the burner J, which has a pilot light, or else to the apparatus under test, which is attached to M. The operation is thus continuous, the gas mixture being used at the same rate at which it is prepared either by burning at J or in the test apparatus, both branches being adjusted to give the same rate of flow. This rate of flow is adjusted to suit the pressure in the mixing bottle by means of the cock K, placed just before the three-way cock L. A small portion of the gas can be withdrawn for analysis through the tube N, to which is attached a five-bulb absorption tube and a wet meter in series (not shown in figure).

When starting a run, the burette, filled with ether in which the amount of dissolved hydrogen sulphide has been determined, is placed in position and adjusted so that the solution drops at a uniform rate. The gas flow is also properly adjusted, and the

gas mixture burned at J for 45 to 60 minutes to insure a uniform and constant mixture in the mixing bottle. When tests with lead acetate paper, made from time to time, show that this condition has been reached, the mixing operation is stopped so that the burette may be detached and weighed and the meter reading taken; the mixing is then again started. The weight of the burette and the meter reading are again obtained at the end of the run. From the weight of hydrogen sulphide solution introduced and its strength and the volume of gas plus ether through which it has been distributed, the hydrogen sulphide content of the gas can easily be calculated. A small portion of the gas is withdrawn through N, and the contained hydrogen sulphide absorbed in a solution of cadmium acetate.

The precipitated cadmium sulphide is then determined iodometrically. The determination of the hydrogen sulphide in the ether solution and in the gas is fully discussed on page 36 ff.

2. METHOD OF COMPARISONS

Having available a gas mixture, the hydrogen sulphide content of which could be controlled and kept uniform for any desired period up to several hours, we were able to make tests in which any one factor could be changed at will, while all others were maintained constant. Comparisons of sensitivity of one procedure with another were, therefore, easily obtained with a gas of any desired strength.

Since preliminary work indicated that the simple hydrogen sulphide tester (see Fig. 10) which was later chosen as standard, had a number of advantages over the other forms investigated, this apparatus was used in the study of the variables introduced by changes in the paper and in the gas, groups 1 and 2 indicated on page 11. When various forms of apparatus were being compared, they were connected in the set-up in parallel in such a way that the gas could be diverted to one or another at will. Comparisons between one procedure and another could thus be made very rapidly. No effort was made to have the same quantity of hydrogen sulphide present in the gas on different days. It is therefore not intended that comparisons should be made from one chart to another.

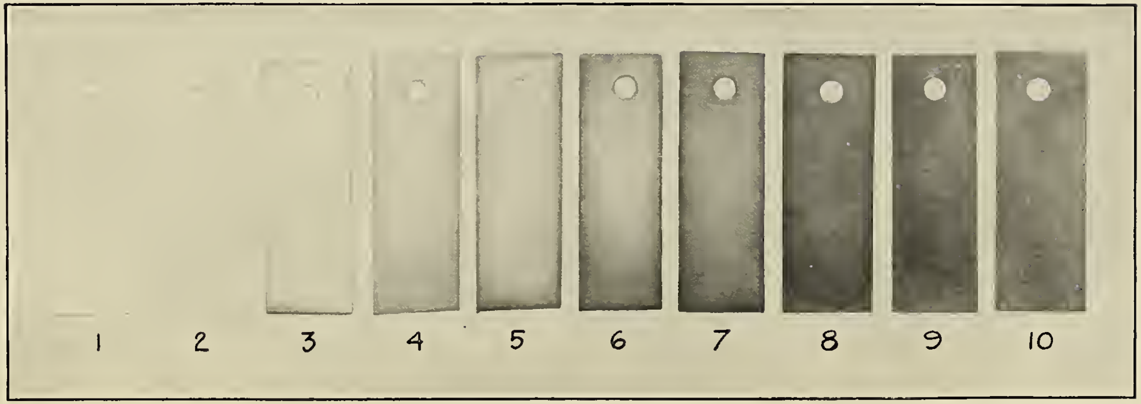

Fig. 2.—*Standard color chart*

To insure the uniformity of the gas supply throughout the period of any one series, control tests were run. These tests were made from time to time, and practically no difficulty was found in maintaining the mixture so uniform as to give exactly comparable results throughout any series.

3. RECORDING OF RESULTS

The problem of recording results offered a number of difficulties, the most serious of which was due to the impossibility of giving an accurate and brief verbal description of a test paper after exposure. The uncertainty which any such description leaves in the mind of a reader is well illustrated by the results reported by Carpenter. (See p. 8.) Therefore a photographic record of the tests was made. Test papers illustrated in any one plate are generally comparable; but care must be exercised in comparing the test papers shown in the different figures, because the proper relative depth of coloration is very difficult to secure in illustrations made by photographic processes. It should be particularly noted that a very light-brown coloration may appear quite dark when photographed, and may even appear to be black. On the other hand, some papers, showing a denser but silvery coloration, reflect more light than others less dense but of a brownish character; and, consequently, when photographed, the former may appear relatively too light.

To make it possible to record approximately the depth of coloration produced in any test, and thus give a basis for comparison of tests made on different days, a standard color chart was prepared. (See Fig. 2.) In making this chart a series of ten test papers was selected to form a graduated scale of colors, ranging from No. 1, which is a blank, not having been exposed to the gas, to No. 10, which is a very dark brown, so heavily stained that it shows some tendency to appear silvery and, hence, appears much lighter in color, relatively, than otherwise. Standard color No. 2 was selected as having as faint a coloration as could be easily distinguished without comparison with a blank paper.

Since test papers fade somewhat on keeping, those used for the standard-color chart were selected from a lot which had already aged for some time, so that further changes in color would be

slight and would have but little influence on the usefulness of the chart as a means of comparing results. The colors were selected strictly empirically and have no significance as representing definite amounts of hydrogen sulphide, except as the tests described below give some significance to them.

As tests were made the papers were matched as closely as possible with the standard colors and then dried in a dessicator in order to reduce fading to a minimum. When a series had been completed the test papers were mounted on cardboard and photographed. When thus mounted, differences in depth and character of the colors could be distinguished more easily than when unmounted, since the papers lay flat and there were no interfering shadows. This system of recording results proved very satisfactory.

V. VARIATIONS IN TEST PAPER

1. KIND OF PAPER

The kind of paper used is not without influence on the accuracy of the test. Among the factors that affect the ease with which slight differences in color may be distinguished are: The tint of the paper, the character of its surface, whether it is smooth or matte, the thickness and opacity, and the change in opacity brought about by moistening.

The most satisfactory paper to use is a hard, smooth, and fairly opaque filter paper of even thickness. Schleicher and Schüll's hardened filter No. 575 was found to be most satisfactory for our purposes and was used in making all tests. Test papers of this kind have the same general appearance when moist as when dry, so that a moist paper could be compared with the standard colors conveniently. None of the other papers tested was satisfactory in this respect.

The difference in appearance noted, when the same gas was tested in the same manner with different kinds of paper similarly prepared, is illustrated in Fig. 3 and described in Table 3.

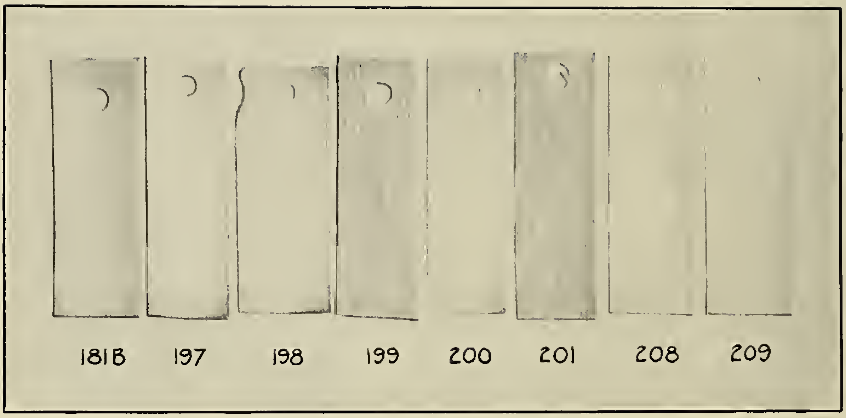

FIG. 3.—*Appearance of tests made on different kinds of paper*

(Described in Table 3)

TABLE 3

Appearance of Tests Made on Different Kinds of Paper

(Illustrated in Fig. 3)

[All tests were made with moist paper, which had been prepared by dipping in 6.5 per cent solution and blotting, using the standard apparatus, with gas flowing at 5 cubic feet per hour for one minute.]

Test	Description of paper
181B	S. and S. "No. 575 hardened"; opaque with hard, smooth surface.
197	S. and S. "No. 602 extra hard"; surface rougher than No. 575.
198	S. and S. "No. 595"; lighter and softer than either of the above.
199	Rough qualitative filter paper with irregular, ribbed surface.
200	Smooth tablet paper; not as absorbent as the filter papers.
201	Paper towel with faint greenish tinge.
208	S. and S. "No. 575 hardened"; soaked in lead acetate for two minutes, otherwise same as for test 181B.
209	S. and S. "No. 589 blue band"; a thin soft paper, hard to handle without tearing when moist.

Greater differences in the appearance of the papers were noticed than are indicated by the figure and considerable change in the coloration of some of the papers occurred on drying. Only a few generalizations as to the kind of paper are needed, since each observer should satisfy himself that the paper used is suitable for the work to be done.

The following points, although not of great importance, are worthy of mention as a guide in the choice of paper:

1. Any paper having a yellowish tinge tends to obscure the first appearance of a stain when comparison is made with an unexposed piece of the same paper.

2. Papers with a rough matte or wrinkled surface are not as satisfactory as those with a smooth surface, since the rough surface adds an element of uncertainty to the test; but a shiny surface is not desirable.

3. Test papers 197, 198, and 199, which are typical of those made on filter paper with a rough surface, appeared slightly darker than paper 181 when moist, but lighter after they had dried out. This change in appearance on drying seems characteristic of the rough papers.

4. A transparent paper of uneven thickness, if viewed against certain backgrounds when moist, shows more of a coloration than a less transparent paper similarly treated.

5. When there is question as to the result of a test, the test paper should be compared with a piece of paper which has been similarly treated, but not exposed to the gas, the comparison being made with the papers in a good light and held against a white opaque background.

2. CONCENTRATION OF LEAD ACETATE SOLUTION

Results of tests made to determine the effect of varying the concentration of lead acetate solution with which the paper is treated are illustrated in Fig. 4, and the details of the tests are given in Table 4.

TABLE 4

Effect of Concentration of Lead Acetate Solution

(Illustrated in Fig. 4)

[All tests were made with paper exposed in the standard apparatus with gas of approximately 0.7 grain hydrogen sulphide per 100 cubic feet of gas. Paper B was dipped in the lead acetate solution, blotted, and used moist; paper D was dipped, dried, and used dry.]

Tests with paper B				Tests with paper D			
Test	Time (minutes)	Concentration of solution (per cent of lead acetate)	Color produced (standard color No.)	Test	Time (minutes)	Concentration of solution (per cent of lead acetate)	Color produced (standard color No.)
83	0.5	2		84	0.5	2	
85	0.5	6.5	3-4	86	0.5	6.5	2
87	0.5	12		88	0.5	12	
89	1	2		90	1	2	
91	1	6.5	5-6	92	1	6.5	3
93	1	12		94	1	12	
95	3	2		96	3	2	
97	3	6.5	6-7	98	3	6.5	5-6
99	3	12		100	3	12	

The papers illustrated in the three horizontal rows of this figure, which were exposed for one-half, one, and three minutes, respectively, show the effect of increasing time on the depth of color produced. However, the first three papers of each row, which were treated with 2, 6.5, and 12 per cent solutions of lead acetate, respectively, then blotted, and used moist, show how small an effect is produced by changing the concentration of the solution. The second three papers, which were treated in the same manner as the first three, except that they were dried before use, also show this effect to be very small.

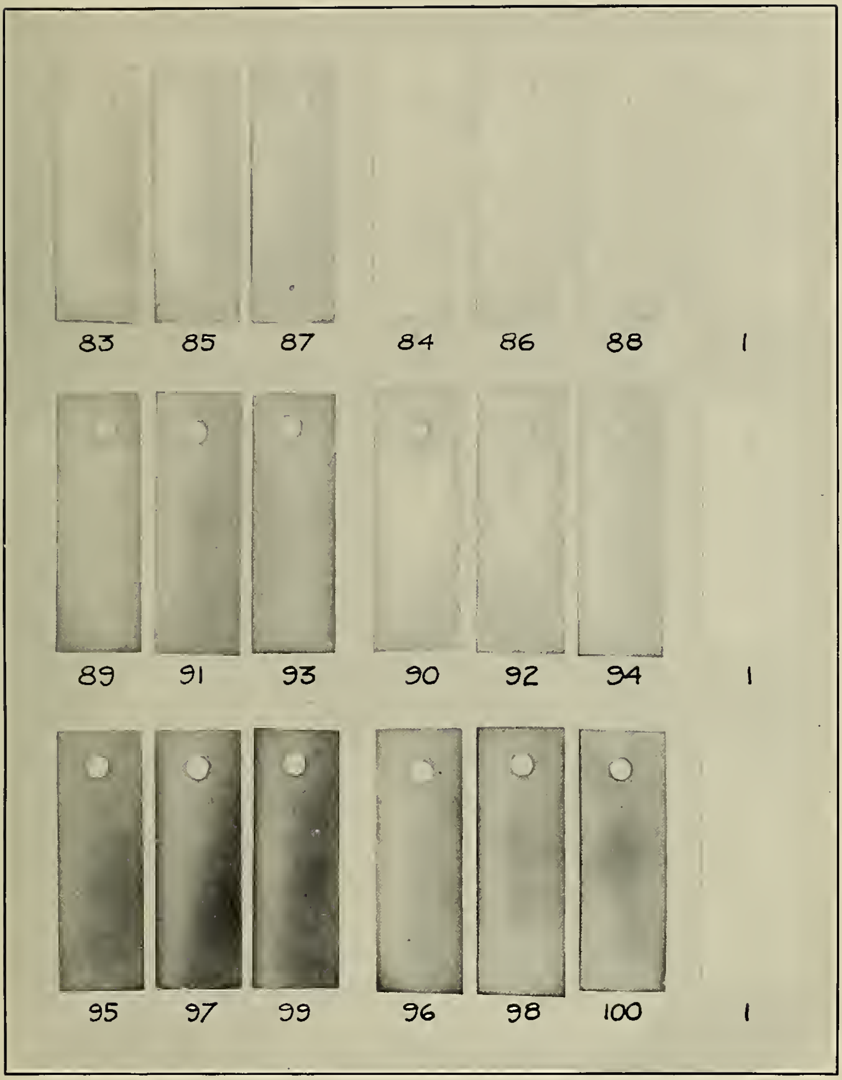

Fig. 4.—*Effect of concentration of lead acetate solution*

(Described in Table 4)

Since the paper, even with the 2 per cent solution, holds a large excess of lead acetate over that necessary to precipitate the hydrogen sulphide coming in contact with it, it is not surprising that little difference is to be noted in the sensitivity of the test when different concentrations of lead acetate solution are used, although there seems to be a tendency for the paper treated with the 2 per cent solution to be slightly lighter in color than the other two.

Dibdin and Grimwood from a comparison of "papers soaked in various strengths of lead salts in solution * * * decided to adopt a paper prepared by moistening with an approximately 12 per cent solution of lead acetate." They do not state their reason for the choice, and the above-described results give no basis for any preference within the limits investigated.

The use of a 6.5 per cent solution corresponds to both American and English practice, arising from the old English rule which called for 100 grains of crystallized lead acetate dissolved in 100 cc of water. The 6.5 per cent solution was used for the bulk of the experimental work presented in this paper because of its previous wide use. However, a 5 per cent solution would give the same results and its use is recommended.

3. PREPARATION AND CONDITION OF PAPER

Papers dipped in a 6.5 per cent solution of lead acetate were treated in eight different ways, in order to ascertain the effect of varying preliminary treatment and moisture content at time of use. The papers used are described in the following tabulation, which also indicates the letter by which each paper is identified in the description of later tests.

A. Dipped and used wet.

B. Dipped, blotted, and used moist.

C. Dipped, dried in air, preserved over water.

D. Dipped, dried in air, preserved over calcium chloride.

E. Dipped, dried in an atmosphere of carbon dioxide, preserved over calcium chloride.

F. Dipped, dried in an atmosphere of carbon dioxide, preserved over water.

G. Dipped, dried in an atmosphere containing a large amount of ammonia, preserved over calcium chloride.

H. Dipped, dried in an atmosphere containing a large amount of ammonia, preserved over water.

The papers which were dipped and used without blotting (e. g., paper A) of course had a considerable amount of water adhering to them. They are designated as "wet," whereas those blotted before use (e. g., paper B) are termed "moist." This distinction between "wet" and "moist" is followed throughout this paper. The papers preserved in a desiccator over calcium chloride were, of course, thoroughly dry when used, while those kept over water contained enough moisture to be detected by touch. The difference in sensitivity of papers preserved over water and those preserved over calcium chloride is of importance as showing the possible variation of test results obtained when papers are kept under varying natural atmospheric conditions.

It is not an uncommon practice to prepare test papers and preserve them in contact with the air for some time before use, giving opportunity for absorption of carbon dioxide from the air by the lead salt in the paper. It was desirable, therefore, to determine what effect this might have on the sensibility of the papers. To accomplish this papers E and F were prepared.

It has been stated by a number of observers that exposing the test paper to ammonia vapor makes it more sensitive, but no conclusive experiments on this point had been reported. To test the truth of this contention papers G and H were prepared and used.

In the preparation of papers which are to be used dry, large sheets should be dipped in the lead acetate solution and then dried. The edges of these sheets should be trimmed off before cutting them up into the smaller test pieces. When the individual test papers are dipped and dried they are found to have a mottled appearance after exposure, the color appearing in irregular spots, particularly along the edges. Papers prepared by the first method give a more even distribution of the color.

The tests of these papers are illustrated in Fig. 5 and the details of the tests are given in Table 5. As in the previous set, the papers illustrated in the first row were exposed one-half minute, those of the second row one minute, and those of the third row three minutes. Papers prepared and used in the same manner appear in the same vertical column.

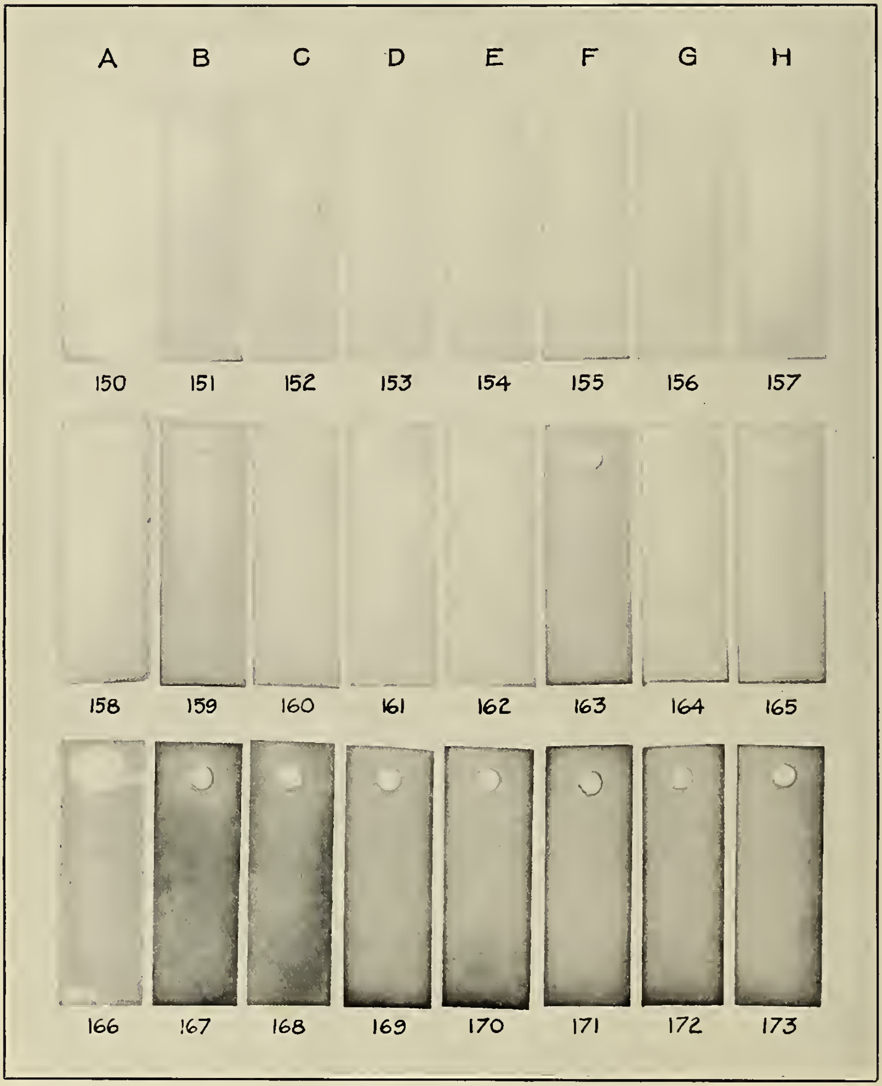

FIG. 5.—*Effect of preparation and condition of test paper*

(Described in Table 5)

TABLE 5

Effect of Preparation and Condition of Test Paper

(Illustrated in Fig. 5)

[All tests were made in the standard apparatus, with gas containing approximately 1.1 grains hydrogen sulphide per 100 cubic feet.]

Test	Paper	Time	Color produced (standard color No.)	Remarks
150	A	0.5	4−	Mottled.
151	B	0.5	3	
152	C	0.5	3+	
153	D	0.5	2−	
154	E	0.5	2+	
155	F	0.5	4	
156	G	0.5	2	
157	H	0.5	3−4	
158	A	1	5	Silvery appearance.
159	B	1	6−	
160	C	1	5	
161	D	1	4−	
162	E	1	4−	
163	F	1	6	
164	G	1	3−4	
165	H	1	5+	
166	A	3	8	Silvery.
167	B	3	8	Uniform color distribution.
168	C	3	7	
169	D	3	7+	
170	E	3	7	Darker than standard at edge.
171	F	3	7+	Black edges.
172	G	3	7	Black edges.
173	H	3	7	Dark edges, brown.

It is at once apparent that the moist papers are more sensitive than the dry papers. This will be discussed fully in a later section (see p. 24), where even a more striking example of the same effect is shown. The wet paper A, which is prepared by dipping and using without blotting, has an appearance very different from the other papers when it is exposed. It carries a surface film of lead acetate solution in which the hydrogen sulphide is precipitated, giving it a silvery appearance very unlike the other papers where the sulphide is precipitated on the paper fiber. This surface film, while wet, is easily removed by shaking or touching; and when dried it collects in patches and appears considerably lighter than when wet. Notice paper No. 166.

In comparing the moist papers B, C, F, and H, no decisive difference in their sensitivity was noted, at least none which might not be attributed to differences in individual test papers or unavoidable variations in the flow of gas over the test paper. Paper F, which had been dried in an atmosphere of carbon dioxide, seemed to be more sensitive in one or two tests than paper B; but this difference was not important and could not be definitely ascribed to the treatment with carbon dioxide.

No definite difference in sensitivity as compared with papers D and B was noted, either in this series or in a similar series, not illustrated, in the case of papers G and H, which had been treated with ammonia. Since there was no ammonia present on these papers when they were used, it was desired to test in another series the effect of free ammonia. To do this, papers, after being dipped and blotted, were held over concentrated ammonium hydroxide for one minute and then used at once. The coloration of papers so treated appeared to be somewhat lighter with gas of the strength used than the coloration of papers not exposed to the ammonia. These tests are illustrated in Fig. 6 and described in Table 6. Still other tests, not illustrated, were made on papers dipped in concentrated ammonium hydroxide solution, blotted, and used moist; these also showed a tendency to give less coloration, as compared with paper B.

TABLE 6

Effect of Ammonia on Sensitivity of Test Paper

(Illustrated in Fig. 6)

[All tests were made in the standard apparatus with gas containing approximately 1.1 grain hydrogen sulphide per 100 cubic feet.]

Test	Paper	Time (minutes)	Color produced (standard color No.)	Remarks
159A	B	1	6−	
174	B+NH_3	1	5−	Held one minute over concentrated NH_4OH.
175	B+NH_3	1	4-5	Held one minute over concentrated NH_4OH.

It is possible that the increase in sensitivity upon treatment with ammonia, which other authors have recorded, has been

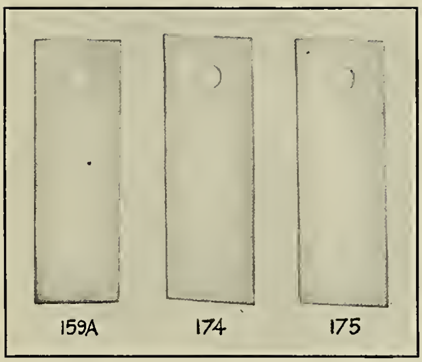

FIG. 6.—*Effect of ammonia on sensitivity of test paper*

(Described in Table 6)

really due to a comparison of dry paper with a paper which, during treatment with ammonia, had taken up considerable moisture. The increase in sensitivity which they noted should, therefore, probably be ascribed to moisture rather than to the ammonia.

4. SUMMARY AND RECOMMENDATIONS

An operator, giving consideration to the desired qualities noted above, can easily select the most suitable and convenient kind of paper after trying a number of samples, the convenience, but generally not the accuracy, of the test being affected by the choice.

There is little or no difference in the sensitiveness of paper prepared with lead acetate solution from 2 to 12 per cent in strength; a 5 per cent solution is recommended. Treatment of the papers with ammonia or carbon dioxide does not materially change their sensitiveness.

For reasons, some of which are given in the preceding section and others of which are made clear in the later discussions, the use of paper prepared in the same manner as paper B, i. e., dipped, blotted, and used moist, is recommended; such paper gives the most reliable and most nearly reproducible results.

VI. VARIATIONS DUE TO THE GAS

1. HUMIDITY OF THE GAS

The humidity of the gas and the condition of the paper, whether moist or dry, are factors which have a very decided influence on the sensitiveness of the test. This is strikingly shown by a series of tests on "dry" and "wet" gas with both moist (B) and dry (D) papers. In the first group of these tests the gas was dried after leaving the meter by passage through a tall calcium chloride tower (D of Fig. 1). When the tests on the dry gas had been completed, the connection to the drying tower was shut off and the gas, saturated at room temperature with water vapor by passage through the wet meter, was used directly.

The results obtained are tabulated in Table 7 and the test papers are shown in Fig. 7. The papers illustrated in the first horizontal row were used moist and the papers in the second row were

used dry. The first four vertical rows show tests made with dry gas and the second four with wet gas.

TABLE 7

Effect of Humidity of the Gas

(Illustrated in Fig. 7)

[All tests were made in the standard apparatus with gas containing approximately 0.7 grain hydrogen sulphide per 100 cubic feet.]

Dry gas				Wet gas			
Test	Paper	Time (minutes)	Color produced (standard color No.)	Test	Paper	Time (minutes)	Color produced (standard color No.)
67	B	0.5	3	75	B	0.5	3
69	B	1	4	77	B	1	4
71	B	3	6	79	B	3	7
73	B	5	6–7	81	B	5	8
68	D	0.5	1	76	D	0.5	2–
70	D	1	1+	78	D	1	3
72	D	3	1+	80	D	3	6
74	D	5	1+	82	D	5	7

It will be noted that the dry paper in the dry gas (Tests No. 68, 70, 72, and 74) gives only the faintest perceptible tinge even in five minutes, whereas the dry paper in the wet gas gave a distinct test in one-half minute (Test No. 76) showing the effect of the wet gas in moistening the paper. Practically the same test is obtained with moist paper when exposed for one-half or one minute in either the wet or dry gas; but when exposed for five minutes the strongest test is obtained with the wet gas, showing the drying effect of the dry gas. Although the moist paper exposed to the dry gas dries out very quickly and loses its sensitiveness, still it is more sensitive for short periods in the dry gas than the dry paper in the wet gas. On the other hand, when exposed for three to five minutes, the dry paper becomes sufficiently sensitive in the wet gas to give the same test as the moist paper exposed in the dry gas for the same length of time.

The use of dry test papers is not recommended, since the humidity of the gas may vary widely, being quite low whenever the gas has been subjected to low temperatures, thus making the

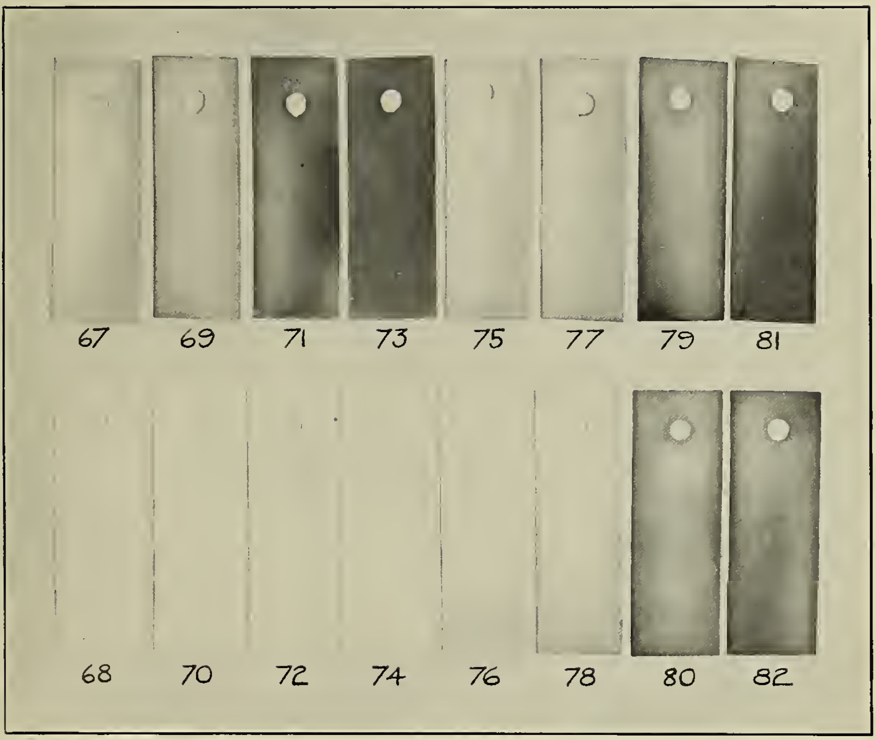

FIG. 7.—*Effect of humidity of the gas*

(Described in Table 7)

test variable and more dependent on the moisture content of the gas than on its hydrogen sulphide content. The moist paper is not "unduly sensitive" and it is evident that it will give the most reliable indications in practice.

2. RATE OF FLOW OF THE GAS

It is obvious that the depth of coloration obtained in any test depends not only upon the amount of hydrogen sulphide in the gas, but also upon the amount of the gas coming in contact with the paper. The time of exposure and the rate of flow of the gas will in part determine the amount of gas coming in contact with the paper. The effect, however, is not usually proportional to the quantity of gas passing; that is, the same test will not be obtained by exposing a paper for one minute to a gas flowing at the rate of 5 cubic feet per hour as by exposing the same paper to the same gas flowing at the rate of 1 cubic foot per hour for five minutes, although the same amount of gas flows through the apparatus in both cases. The coloration produced increases about proportionally with the increase in the time of exposure, but an increase in rate of flow gives much less than a proportional increase in coloration.

These effects of varying the rate of gas flow and the time are shown by the tests recorded in Table 8 and Fig. 8. When making tests at different rates, by-passes were so arranged that the apparatus under test and the by-pass in parallel with it at any time consumed a total of 5 cubic feet per hour, in order that the adjustment of the mixing apparatus would not be disturbed by changes in the rate of flow of the gas to the test apparatus. The by-passes were adjusted and calibrated in advance and were so arranged that a change in the gas rate could be made instantly. Comparisons could, therefore, be made quickly and without chance for variation in other conditions.

TABLE 8

Effect of Rate of Flow of Gas

(Illustrated in Fig. 8)

[All tests were made in the standard apparatus with gas containing approximately 0.5 grain hydrogen sulphide per 100 cubic feet.]

Test	Paper	Gas rate (cubic feet per hour)	Time (minutes)	Color produced (standard color No.)	Remarks
1	Blank	....	....	....	
180	B	5.0	0.5	2-3	
181	B	5.0	1	3-4	
182	B	5.0	3	6	
189	B	3.5	0.5	2	
190	B	3.5	1	3−	
191	B	3.5	3	5	
192	B	0.8	0.5	1	
193	B	0.8	1	1+	
194	B	0.8	3	4−	
186	B	5.0	0.5	2+	Watch glass out (see p. 32).
187	B	5.0	1	3+	
188	B	5.0	3	6+	
181A	B	5.0	1	3-4	"Control" test (see p. 15).
195	B	....	45	4-5	See p. 33.
196	B	5	3	1+	Referees apparatus.
183	D	5.0	0.5	1-2	Paper used dry.
184	D	5.0	1	2-3	
185	D	5.0	3	5−	

The lighter coloration produced at the lowest rate is quite apparent. The difference between the 3.5 and the 5.0 cubic feet per hour rates is not so noticeable; practically the same results would be obtained between the limits of 4.5 and 5.5 cubic feet per hour, and these limits were chosen for the method recommended.

3. TIME OF EXPOSURE

As pointed out in a previous paragraph, the time of exposure is very important in determining the depth of coloration produced, the sensitiveness of a test being roughly proportional to the time. However, attention may be called again to the tests made with dry paper in dry gas, where there was little difference noticeable between tests of one-half and five minutes' exposure. The proportionality above noted, however, holds good when the test paper is moist, unless a large amount of hydrogen sulphide is

FIG. 8.—*Effect of rate of flow of gas*

(Described in Table 8)

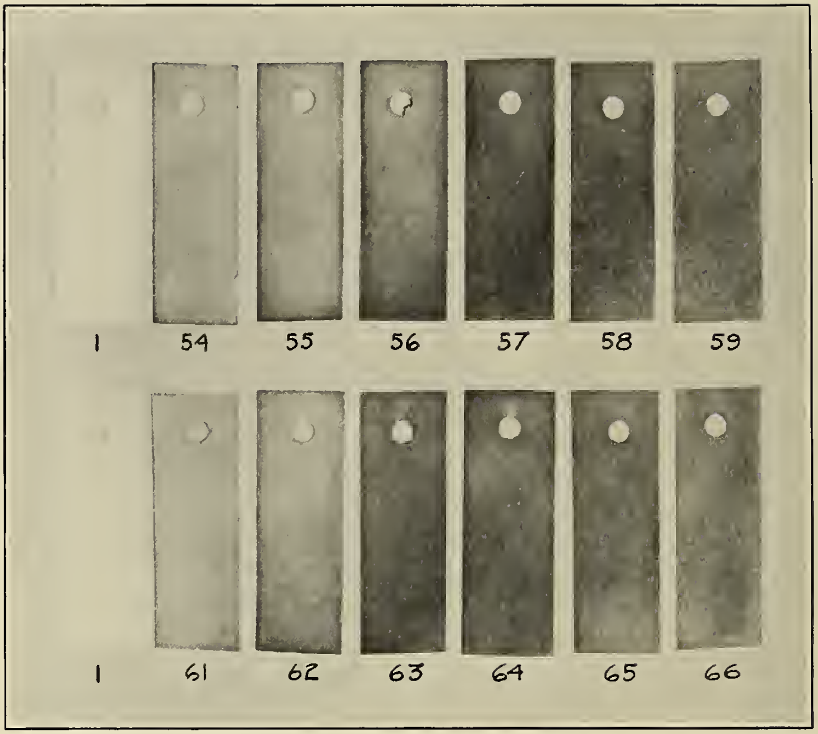

FIG. 9.—*Effect of time of exposure*

(Described in Table 9)

present in the gas. In this latter case the depth of coloration does not increase so markedly with the time; this case is illustrated in the tests reported in Fig. 9 and Table 9. The papers exposed more than one minute show only slight increase in color, especially in the photographic reproductions, since the character of the coloration is changed slightly, becoming silvery. This is particularly true of the tests made with paper H, which had been treated with ammonia.

TABLE 9

Effect of Time of Exposure

(Illustrated in Fig. 9)

[All tests were made in the standard apparatus with gas containing approximately 2.7 grains hydrogen sulphide per 100 cubic feet.]

Test	Paper	Time (minutes)	Color produced (standard color No.)	Test	Paper	Time (minutes)	Color produced (standard color No.)
1	Blank	....	....	1	Blank	....	
54	B	0.25	5	61	H	0.25	
55	B	0.5	6–7	62	H	0.5	Correspond
56	B	1	8	63	H	1	closely to
57	B	2	8–9	64	H	2	Nos. 54–59.
58	B	3	9	65	H	3	
59	B	4	9–10	66	H	4	

The variation in test results with time is illustrated in a majority of the other figures, and these should be noted in this connection.

4. SUMMARY AND RECOMMENDATIONS

The large effect of the humidity of the gas upon the test results is shown, but it is also noted that the use of moist paper with short test periods (not over three minutes) gives results practically independent of the humidity of the gas. It is impracticable either to dry or to saturate the gas to a constant condition as to humidity, because of the probability of simultaneous change in the hydrogen sulphide content.

The rate of gas flow is shown to have some effect, but increases in rate produce less effect than proportional increases in the time of test. The general practice of making tests at 5 cubic feet per hour is satisfactory; maintenance of the rate within the limits 4.5 and 5.5 cubic feet per hour is sufficient.

The time of test is an important factor in determining its sensitivity. The proper basis for selection of the length of test will be treated in connection with the discussion of the quantitative and engineering significance of the tests.

VII. FORMS OF TESTING APPARATUS

The different forms of testing apparatus used may be classified into three groups—circulating, impinging, and penetration—these being distinguished by the manner in which the gas is brought into contact with the paper. The five forms of circulating apparatus tested were: The simple hydrogen sulphide tester, chosen for this work and designated in this paper as the standard apparatus; the Referees apparatus; the Young's sulphur and ammonia test apparatus; the small drying-tower form; and a large Woulff bottle. Only one very simple form of the impinging type was tested; and a single penetration apparatus, devised by the authors, was used.

Fig. 10.—*Simple hydrogen sulphide tester, designated as "standard apparatus"* (¼ *size*)

1. DESCRIPTION OF APPARATUS

(1) *Simple Hydrogen Sulphide Apparatus.*—The simple hydrogen sulphide tester, which is illustrated in Fig. 10, was devised in order to secure an apparatus which would be simple and inexpensive in form and such that it would permit rapid, easy, and accurately reproducible test results to be obtained. Although it follows other forms in many details, it has the advantage that it can be easily and cheaply made from materials obtainable in almost any chemical laboratory or drug store without the sacrifice of the advantages due to exactness of specifications.

This apparatus is made from a glass tube, or a common cylindrical gas chimney, 20 cm (8 inches) long and 4.5 cm (1.75 inches) in diameter, closed at top and bottom by perforated stoppers.

The pillar of a gas burner, from which the lava tip has been removed, is inserted through the lower stopper; and a small watch glass, 2.5–3 cm (1–1.5 inches) in diameter, is supported above the burner, to prevent the gas from impinging directly from the inlet on the test paper. The watch glass is supported on three glass pegs, 1–3 cm (0.4–1 inch) high, being held in place with small bits of wax. The gas is burned from an ordinary open-flame burner on the upper stopper; this burner should be so selected that it will pass 5 cubic feet of gas per hour under the ordinary pressure

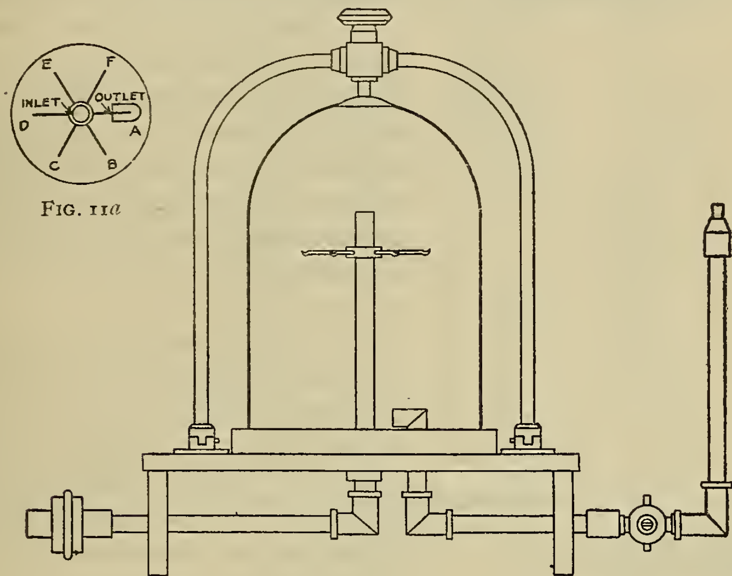

FIG. 11*a*

FIG. 11.—*Referees hydrogen sulphide test apparatus* (½ *size*)

of the gas supply. The test paper, which should be 2 by 6 cm (0.75 by 2.4 inches), is hung on a glass hook so that it is held midway between the watch glass and the bottom of the upper stopper.

(2) *Referees Apparatus.*—This form (see Fig. 11) is one used commonly in this country and for all official testing in London and many other English cities. It consists essentially of a small bell jar held down into a mercury seal, arranged with a central inlet tube and outlet tube with elbow at the base. The test papers are hung on the six small hooks surrounding the inlet tube, as shown in the diagram, Fig. 11*a*.

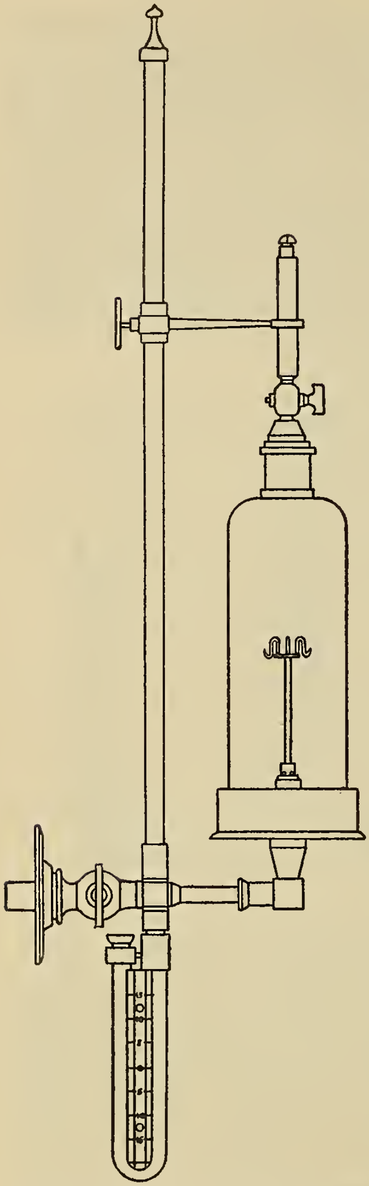

Fig. 12.—*Young's sulphur and ammonia test apparatus* (½ *size*)

(3) *Young's Sulphur and Ammonia Test Apparatus.*—In this form (see Fig. 12) the gas enters the test chamber through openings in the side of the pillar at the base and passes out through the cock and burner at the top. The small bell is taken off to insert or remove test papers by detaching the clamp from the upper pillar.

(4) *Small Drying-Tower Form.*—An ordinary calcium chloride tower (see Fig. 13) the upper chamber of which was about 14 cm (5.5 inches) long and 2.7 cm (1 inch) wide, inside diameter, was arranged by attaching an inlet tube at the base and a stopper with glass hook and burner at the top.

(5) *Large Woulff Bottle.*—This form was arranged as shown in Fig. 14, using a Woulff bottle 19 cm (7.5 inches) tall and 14 cm (5.5 inches) in diameter. The gas entered through the tube extending almost to the bottom and was burned at the burner on the right-hand stopper; test papers were hung on the glass hook in the central stopper. This apparatus was also used for a few tests in which the papers were exposed for 30 to 45 minutes to a sample of gas confined in but not flowing through the bottle.

(6) *Apparatus of Impinging Type.*—The only form of impinging apparatus tested consisted of a glass tube drawn out to a tip, from which the gas impinged directly upon the test papers held in

position at the desired distance in front of the jet. Some of the older forms of apparatus, similar in form to those of the circulating type, were so made that the gas issuing from small orifices impinged directly upon the test papers; the Referees apparatus used from 1877 to 1906 was of this form. None of these older forms of apparatus was tested.

(7) *Penetration Apparatus.*—The penetration apparatus used, which was devised by the authors, is shown in cross section in Fig. 15. The gas enters at the bottom, is forced through the test paper which is held between the two glass parts and is burned from the

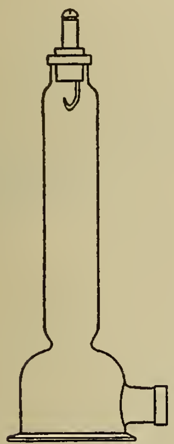

FIG. 13.—*Small drying-tower form* (½ *size*)

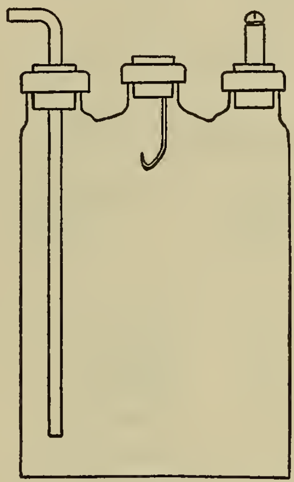

FIG. 14.—*Large Woulff bottle apparatus* (½ *size*)

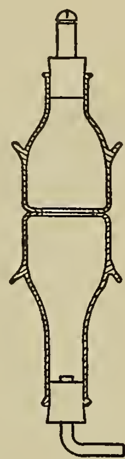

FIG. 15.—*Penetration apparatus* (½ *size*)

burner at the top. The two halves of the apparatus are held together firmly by strong rubber bands between the glass hooks shown at the sides. The opening covered by the test paper is 1 inch in diameter; and the faces of the two sections are ground plane, so that there is a gas-tight connection when the rubber bands are in place. It was necessary to use only dry test papers in this apparatus, since moist paper is not sufficiently porous. The apparatus passed about 2.5 cubic feet of gas per hour and was used in parallel with a burner consuming the same amount, so that when substituted for other forms of apparatus it caused no change in the total gas rate.

2. COMPARISON OF APPARATUS

The results of a comparison of the five forms of circulating apparatus are given in Fig. 16 and Table 10. These tests show that the darkest coloration is produced in the small drying tower and the lightest in the Referees apparatus. Arranged in order of decreasing sensitiveness of test obtained, the forms of apparatus tested are as follows: (1) Small drying tower, (2) Young's sulphur and ammonia tester, (3) standard apparatus, (4) Woulff bottle, and (5) Referees apparatus. Another series of tests with dry paper showed the same results.

TABLE 10

Comparison of Apparatus

(Illustrated in Fig. 16)

[All tests were made with gas containing approximately 0.6 grain hydrogen sulphide per 100 cubic feet.]

Test	Paper	Time (minutes)	Apparatus	Color produced (standard color No.)
1	Blank			
101	B	0.5	Standard	1-2
102	B	0.5	Drying tower	3−
103	B	0.5	Woulff bottle	1+
104	B	0.5	Referees	1
105	B	0.5	Young's	2+
111	B	1	Standard	3
112	B	1	Drying tower	4
113	B	1	Woulff bottle	1-2
114	B	1	Referees	1
115	B	1	Young's	3
121	B	3	Standard	5
122	B	3	Drying tower	8−
123	B	3	Woulff bottle	4
124	B	3	Referees	1-2
125	B	3	Young's	6

Tests No. 186, 187, and 188, given in Fig. 8 and Table 8, show the effect of operating the standard apparatus without the usual watch glass above the inlet. By comparison with No. 180, 181, and 182, it will be noted that the coloration is slightly stronger when the watch glass is removed.

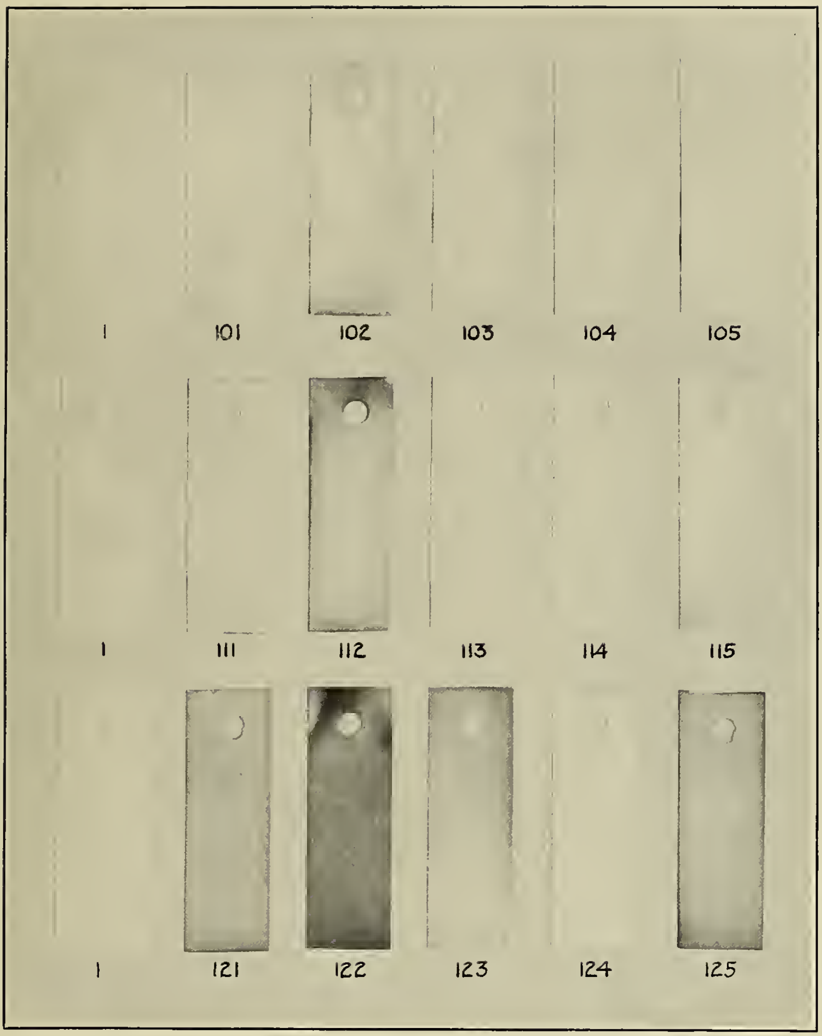

FIG. 16.—*Comparison of apparatus*

(Described in Table 10)

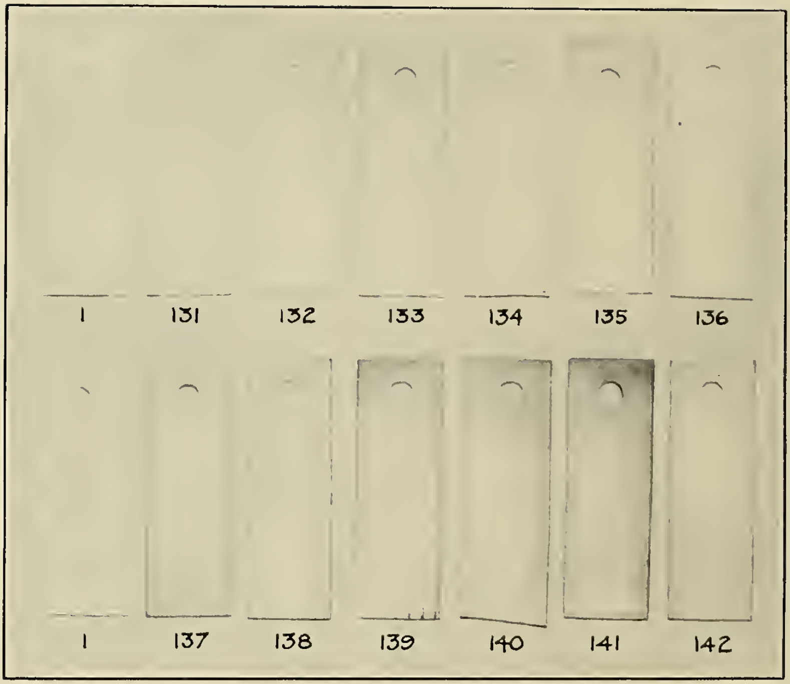

FIG. 17.—*Position of paper in Referees apparatus*

(Described in Table 11)

In the same table and figure is described a test, No. 195, in which a moist paper, B, was hung for 45 minutes in the Woulff bottle (Fig. 14) previously filled with gas containing 0.5 grain of hydrogen sulphide per 100 cubic feet.

The Referees apparatus is not symmetrical, the gas outlet being at the bottom near one side. A difference in the test obtained is noticed, depending upon which of the six hooks holds the test paper. The arrangement of these hooks with reference to the outlet is shown diagrammatically in Fig. 11*a*, the six possible positions for the test paper being lettered A to F. Two tests were made with a test paper suspended from each of the six hooks; the results are given in Fig. 17 and Table 11. In the three-minute test, shown in the upper row of the figure, the papers in positions A, B, and F show a very faint coloration; but the papers in positions C and E, and to a slightly less extent D, were more distinctly colored, particularly at their upper ends. The same differences are to be noted in the 10-minute test, the papers suspended in positions C, D, and E being darkest.

TABLE 11

Position of Paper in Referees Apparatus

(Illustrated in Fig. 17)

[Paper B was used in these tests, with gas containing approximately 0.6 grain of hydrogen sulphide per 100 cubic feet.]

Time, 3 minutes			Time, 10 minutes		
Test	Position of paper	Color produced (standard color No.)	Test	Position of paper	Color produced (standard color No.)
1		Blank	1		Blank.
131	A	1+	137	A	2+
132	B	1+	138	B	2
133	C	2	139	C	3
134	D	1–2	140	D	2–3
135	E	2	141	E	3
136	F	1+	142	F	2

Only a few tests were made with apparatus of the impinging type; these showed very clearly the increase in sensitiveness when the gas was allowed to impinge on the paper from a jet. A moist paper, held 0.5 mm from the tip of a jet, from which the gas

issued at the rate of 1 cubic foot per hour, showed in half a minute a coloration that was just barely perceptible, but in one minute a distinct coloration was noted; whereas with the standard apparatus the same gas, at the rate of 5 cubic feet per hour, produced no coloration in five minutes. This gas contained approximately 0.1 grain hydrogen sulphide per 100 cubic feet (1 in 630 000 by volume). Increasing the rate of gas flow increased the sensitiveness of the test somewhat; and increasing the distance from jet to paper decreased the sensitiveness.

Since only apparatus of the circulating type is of importance in practical gas testing, very few tests were made on the other forms.

In Fig. 18 and Table 12 are given the results of one series of tests on the penetration apparatus. Tests with this apparatus are, of course, more sensitive than those made with any of the circulating type, since all of the gas is brought into intimate contact with the paper. The penetration apparatus may be useful when it is desired to have an extremely sensitive test, but for general inspection work it is not recommended.

TABLE 12

Tests with Penetration Apparatus

(Illustrated in Fig. 18)

[All tests were made with paper D and with gas containing approximately 0.6 grain hydrogen sulphide per 100 cubic feet flowing at 2.5–3.0 cubic feet per hour.]

Test	Time (minutes)	Color produced (standard color No.)	Remarks
144	1	5	Upper side of paper is shown, illustrating penetration of coloration through paper.
145	1	5	Lower side of paper is shown; lower side of 144 would match these.
146	1	5	
147	3	8	Lower and upper sides were about the same color.

3. SUMMARY

The variation in the tests with different forms of apparatus is largely due to the variation in intensity of the gas currents and their direction in relation to the paper. In the small drying tower the test paper was suspended close to the rubber stopper bearing the burner and a much larger proportion of the gas came in con-

FIG. 18.—*Tests with penetration apparatus*

(Described in Table 12)

tact with the paper than in the other forms of apparatus. Although the Woulff bottle had a larger capacity than the Referees apparatus, it consistently gave a stronger test than the latter, showing that size alone is not a determining factor; the shape of the apparatus and the location of the paper, relative to inlet and outlet, have an important influence.

In the case of the standard apparatus the removal of the watch glass was shown to make the test more sensitive (see p. 26), evidently because the flow of the gas over the surface of the paper is increased by this change. With the Referees apparatus a gas which was very near the limit might pass inspection when the paper was suspended from one hook and might be condemned when the test was made with the paper in a different position, again showing the variation in the results with differences in gas current over the test paper.

It has been generally recognized that a test in which the gas impinges on the paper is more sensitive than one in which the gas does not impinge. This is due to the better opportunity for contact of the gas with the reagent on the paper and to the smaller area within which the lead sulphide is concentrated. A similar increase in sensitiveness is accomplished by causing the gas to be tested to pass through the paper, as is done in the penetration apparatus described. This increase in sensitiveness, due to the impingement of gas on the paper, seems to have been overestimated by some, however. (See p. 43.)

The important influence of the shape and size of the apparatus upon the result of tests made with it makes clear the necessity of an exact specification of the form and size of the tester. It is therefore recommended that the specifications given for the apparatus which is used as standard in this work be followed exactly in making the tests.

VIII. QUANTITATIVE SIGNIFICANCE OF TEST

When the test is made under definite conditions, the coloration produced is of quantitative significance as indicating the amount of hydrogen sulphide present in the gas. The recognition of the amounts of hydrogen sulphide producing colorations of various intensities is a matter of experience and can not be adequately

described nor presented by photographs. However, the minimum amounts of hydrogen sulphide required to give a positive test under definite conditions can be quite closely approximated. Before discussing the sensitivity of the test, the methods used for the determination of hydrogen sulphide will be described.

1. DETERMINATION OF HYDROGEN SULPHIDE IN MIXTURES

Two methods were employed for determining the hydrogen sulphide content of the gas: First, calculation of the hydrogen sulphide content from the amount and concentration of the ether solution introduced; and, second, analysis of the gas by absorption of the hydrogen sulphide in a suitable medium and determination by titration with iodine. For the sake of brevity, the values secured by these two methods will be characterized as the "calculated" and the "analyzed" values, respectively.

The analysis of the solution of hydrogen sulphide in ether was carried out in the following manner: The tip of the weighed burette containing the solution was inserted into a small Erlenmeyer flask closed with a three-hole stopper. An inlet tube, through which hydrogen could be introduced, reached through a second hole halfway to the bottom of the flask; and an outlet tube in the third hole was sealed to the side tube of an Emmerling tower filled with glass beads so that all of the gas leaving the absorption flask had to bubble through this, in order to catch any hydrogen sulphide escaping absorption in the first flask. Ten cubic centimeters of a 5-per cent solution of cadmium acetate with 15 cc of dilute ammonium hydroxide was placed in the flask and about 30 cc of water with 5 cc of the ammonium hydroxide in the Emmerling tower. About 15 grams of the ether solution was run into the flask, slowly at first because of the rapid volatilization of the ether. The flask was then shaken intermittently for 5 to 10 minutes, while a slow stream of hydrogen was passed through the apparatus. The flask and the Emmerling tower were then washed out; and, after boiling off the ether and cooling the solution, hydrochloric acid was added and the liberated hydrogen sulphide titrated with N/100 iodine solution. The burette was weighed

again to obtain the exact weight of the sample taken and the hydrogen sulphide per gram of ether solution calculated.

In the analysis of the gas, the sample taken through the tube N (see Fig. 1) was bubbled through a neutral solution of cadmium acetate contained in a five-bulb absorption tube and then measured with a wet meter. The bulbs were washed out and the solution was heated to the boiling point to drive off any gases which might react with iodine. After diluting to 250 cc, 15 cc of 1 : 1 hydrochloric acid was added and 10 cc of N/100 iodine solution was run in; crystals of potassium iodide were added and the solution was titrated with N/100 sodium thiosulphate until the yellow color just disappeared. After the addition of 4 cc of fresh starch solution the titration was completed by adding more iodine solution until a pale blue tint appeared. The iodine required for the end point was determined by a blank test.

It should be noted that an ammoniacal or other alkaline solution can not be used without serious error in determining hydrogen sulphide in manufactured gas because of its action on other compounds than hydrogen sulphide. This fact has been overlooked by a number of writers who have used and recommended such solutions. Carbon disulphide reacts with ammonia and alkalies, forming compounds which liberate hydrogen sulphide upon acidifying. The following reactions among others, may take place:

$$CS_2 + 4NH_3 = NH_4CNS + (NH_4)_2S$$
$$3CS_2 + 6KOH = K_2CO_3 + 2K_2CS_3 + 3H_2O$$
$$K_2CS_3 + 2HCl = 2KCl + H_2S + CS_2$$

Absorption of hydrogen sulphide is probably incomplete in acid solution, but the permissible hydrogen ion concentrations have not been investigated. The use of an acetate solution would seem preferable to the use of a chloride solution, since the former can be kept practically neutral.

In order to give an idea of the relative quantities of ether, hydrogen sulphide, etc., employed in these procedures, the data obtained in the preparation of one gas mixture are given.

PREPARATION AND ANALYSIS OF GAS NO. 20

ANALYSIS OF ETHER SOLUTION OF HYDROGEN SULPHIDE

Weight of ether solution taken	15.75 gram	22.30 gram
Iodine solution consumed	3.13 cc	4.29 cc
Iodine solution equivalent to H_2S in 1 gram ether	.20 cc	.19 cc
Strength of iodine solution from standardization, 1 cc equals		0.000158 gram H_2S
		or 0.00244 grain H_2S
∴ 1 gram ether solution contained		0.0000308 gram H_2S
		or 0.000476 grain H_2S

CALCULATED STRENGTH OF GAS

Weight of ether solution taken	62.8 gram
Volume occupied by ether as vapor at 27° C and 759 mm (temperature and pressure in reservoir)	0.739 cu. ft.
Volume of gas taken	7.432 cu. ft.
Total volume of gas mixture	8.171 cu. ft.

Hydrogen sulphide contained $= \frac{62.8 \times 0.000476 \times 100}{8.17}$

$= 0.37$ grain H_2S per 100 cu. ft.

STRENGTH OF GAS DETERMINED BY ANALYSIS

Volume of gas taken for analysis	0.885 cu. ft.
Iodine solution used, corrected for thiosulphate used	1.59 cc
Hydrogen sulphide found (1.59×0.00244)	0.0039 grain
or ($0.0039 \times 100 \div 0.885$)	0.44 grain per 100 cu. ft. of gas

RATE OF GAS FLOW

Total gas used	8.17 cu. ft.
Gas used for analysis	0.88 cu. ft.
Gas to testing apparatus	7.29 cu. ft.
Time of run	90 min.
Rate of gas flow to testing apparatus	4.85 cu. ft. per hour

Considerable difficulty was experienced in securing agreement between the calculated and analyzed values. The former were generally, as in the example cited, about 0.1 grain per 100 cubic feet lower than the analyzed values. This discrepancy, however, was not considered serious, since a difference of 0.1 grain per 100 cubic feet of gas (1 part in 630 000 by volume) makes no material difference in the interpretation of the results for commercial work. Moreover, the exact strength of the gas mixtures was of interest only in determining the quantitative significance of the tests; it has no bearing on the relative results reported in the

previous sections, nor on the discussion of the engineering significance in the later section.

In order to determine, if possible, why the analyzed values were almost invariably higher than the calculated values blank tests were made. In such a test a gas mixture, prepared by using ether which contained no hydrogen sulphide, was passed through the cadmium acetate solution in the usual way. After the usual length of run the solution was treated with iodine in the regular manner; only 0.06 cc of the iodine solution, practically a negligible amount, was required for the blank, showing that the method employed could scarcely be giving too high a result. Numerous other blank tests, made without adding ether to the gas, confirmed this conclusion. To detect any possible escape of hydrogen sulphide from the absorption apparatus, a test was made by passing a gas containing somewhat over 0.5 grain of hydrogen sulphide per 100 cubic feet through the absorption apparatus and then allowing it to impinge upon a moist lead acetate paper at the rate commonly used in analysis of the mixtures (0.6 cubic foot per hour) for 90 minutes. A copious precipitate of cadmium sulphide was obtained, but no stain was produced on the test paper. This shows that only very minute amounts of hydrogen sulphide could have been escaping from the absorber. The combined evidence of these several special tests gives good ground for confidence in the accuracy of the analyzed values.

The failure to get complete absorption of the hydrogen sulphide when adding the ether solution to the cadmium acetate solution was first suspected as the cause of low values for the calculated strength of the gas. However, increasing the time allowed for absorption was found generally to give still lower values, probably because of oxidation of sulphide, perhaps during the heating to expel the ether. Hydrogen was passed through the flask to minimize this oxidation.

These causes of uncertainty and the difficulties of exact sampling and titrating in the presence of ether made it seem probable that some error in the analysis of the ether solution of hydrogen sulphide was the cause of the discrepancy noted. However, to prove the point conclusively would have required an extended investigation

and much more time than the value of the results at this time would justify. The analyzed values were more concordant and were subject to no systematic error in so far as was known; they were, therefore, accepted in preference to the calculated values for use in the following section.

2. ABSOLUTE SENSITIVITY OF TESTS

In the determination of limiting sensitivity of any testing method which depends upon the appearance of a coloration, the personal judgment of the observer enters very largely. In the present case the test for hydrogen sulphide may have either of two objects: First, the detection of the smallest possible amount of this impurity; or, second, the determination as to whether or not a gas contains more than a permissible amount of the impurity. If a test were made in chemical work where the greatest sensitivity was desired, the first detectable coloration would be considered as sufficient to indicate the presence of hydrogen sulphide. However, in commercial work a distinct coloration would be required before hydrogen sulphide would be recorded as "present." The commercial application of this principle is explained more fully in the recommendations given later. In the following statements as to the sensitivity of tests, it is understood that a distinct coloration is required for positive results.

The results obtained in tests made on moist papers exposed for one minute in the standard form of apparatus are of greatest interest, since they represent the results obtained by the procedure which has been accepted as the standard for this work for reasons made clear in the section of recommendations.

From the various results obtained by this standard procedure, which are summarized in Table 13, it is evident that this test, if of one minute duration, would detect 0.5 grain of hydrogen sulphide per 100 cubic feet of gas; but with a gas of 0.25 grain no distinct test would be obtained. It is impossible to fix a limiting sensitivity of this test more specifically than to say that 0.3 to 0.4 grain of hydrogen sulphide per 100 cubic feet of gas is about the limit which would be detected. If the test is of one-half minute duration, it is of course less sensitive, about 0.45 grain of hydrogen sulphide being required to give a test; if it is extended

to three minutes, a gas of about 0.20 grain will give positive results. The variation in sensitivity, which would be produced by other changes in the test procedure, is evident from the numerous tables and figures given earlier in the paper.

It is not necessary to discuss more exactly the quantitative significance of the various combinations of conditions which are illustrated. It is of interest, however, to note again the relative sensitiveness of tests made with different forms of apparatus.

TABLE 13

Quantitative Significance of Tests

[Tests were made with moist paper (B) exposed for the period indicated in the standard apparatus with gas flowing at the rate of 5 cubic feet per hour.]

Test	Gas No.	Time (minutes)	Strength of gas (grains of H_2S per 100 cubic feet	Coloration produced (standard color No.)	Remarks
326	23	1	0.17	1	No coloration; would pass test.
315	21	1	.25	1	Coloration doubtful.
330	26	1	.30	1+	Very faint; might pass.
328	24	1	.32	1+	Slight color on bottom edge of paper; might pass.
308	20	1	.44	2	Would not pass test.
320	22	1	.47	2	Distinct; would not pass.
329	25	1	.72	2+	Very distinctly colored.
316	21	0.5	.25	1	No coloration.
310	20	0.5	.44	1+	Very faint.
322	22	0.5	.47	1+	Coloration doubtful.
327	23	3	.17	1+	Very faint coloration.

The Young's apparatus is slightly more sensitive than the standard form; the Referees apparatus is much less sensitive, for it will scarcely detect 0.5 grain of hydrogen sulphide per 100 cubic feet of gas when used with moist paper for three minutes at a rate of 5 cubic feet of gas per hour. The sensitivity of a test with it varies also with the position of the paper, being greater for the positions on the side opposite the outlet tube.

The penetration apparatus is somewhat more sensitive than any of the forms of circulating apparatus. The sensitivity of the apparatus of the impinging type is variable, depending upon the rate of gas flow, distance of jet from paper, etc.; but under the most favorable conditions apparatus of this type is the most sensitive of all.

It was thought possible, though not probable, that the presence of the ether vapor in the gas mixtures might affect the sensitivity of the lead acetate papers. To determine whether or not such an effect was produced a gas mixture containing 0.45 grain hydrogen sulphide per 100 cubic feet was made with pentane instead of ether as the solvent for the hydrogen sulphide. This gas gave results which checked as closely as could be expected with a mixture of the same strength made with an ether solution. It was concluded, therefore, that the ether vapor in the gas had no effect beyond that produced by the hydrocarbons ("illuminants") already present in the gas.

3. COMPARISON WITH RESULTS OF PREVIOUS EXPERIMENTERS

The results obtained by Dibdin and Grimwood [7] apply only to tests made with the gas impinging from a jet upon the test papers; we have, therefore, only a small amount of data comparable with their results. It is quite certain, however, that somewhat too great a sensitivity has been ascribed to the procedures used by these experimenters. It should be noted that they do not give any information regarding the analytical method employed for the preparation or testing of the gas mixtures used and there are a number of points in their procedure which are open to criticism. In diluting the gas mixture to give a mixture of lower concentration, the residue of the previous mixture was flushed out with gas free from hydrogen sulphide and the reservoir was considered free from hydrogen sulphide when the gas escaping from it gave no test. In view of the fact that a rubber balloon which could easily absorb considerable amounts of hydrogen sulphide was hanging in the reservoir, it is doubtful whether a blank test made while the gas was passing through the apparatus would show conclusively that all hydrogen sulphide had been removed. When making up the more dilute mixtures, the rubber had opportunity to give off considerable amounts of hydrogen sulphide into the gas during the 30 to 45 minutes required for diffusion of the sample introduced. It is very probable that the gas mixtures which they obtained were, for this reason, richer in hydrogen sulphide than they

[7] Loc. cit.

believed. In any event, the evidence which they present is insufficient to show that this was not the case and it is necessary, therefore, to conclude that the results which they have presented are not conclusive as demonstrating the extreme sensitivity which they ascribe to the test. For example, instead of being able to detect 0.0006 grain hydrogen sulphide per 100 cubic feet of gas in a five-minute test, it seems certain that at least 0.01 grain, and probably more, of hydrogen sulphide per 100 cubic feet of gas would be required to give a coloration in a test of five minutes' duration; however, no attempt has been made in the present work to establish exactly the limiting sensitivity of the impingement method. It is desired, therefore, only to point out the probable fact that too great sensitivity has been ascribed in the past to the impingement tests.

The results given by Carpenter in the Alkali Works Inspector's Report[8] are also lacking in details as to the method of preparation and testing of the gas mixtures used. The sensitivity indicated for the mixtures containing from 0.6 to 0.006 grain of hydrogen sulphide per 100 cubic feet of gas is much less than that claimed by Dibdin and Grimwood. Our experience indicates that the conclusions expressed by Carpenter, though perhaps claiming too great sensitivity, are more nearly correct than those given by Dibdin and Grimwood.

The results which are reported by Ramsburg[9] do not indicate in every case all of the details which would be necessary to duplicate the tests exactly, but in so far as comparison has been made between the present work and that reported by Ramsburg very satisfactory agreement has been obtained. The sensitiveness indicated by him for the "present Referees method" is not very different from that which we have obtained; and the other tests indicate corresponding agreement. If any difference was to be noted, it would probably lie in indicating a greater sensitiveness for the tests as made by Ramsburg; but this slight difference could, we believe, be accounted for by the fact that he analyzed the gas mixtures used by a method, that of Hornby,[10] which tends to give

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[8] Loc. cit. [9] Loc. cit. [10] Gas Engineers' Laboratory Handbook.

results slightly too low because an acid absorbing solution is used. In other words, it is probable that a little more than 0.53 grain hydrogen sulphide per 100 cubic feet was present in the gas used by Ramsburg.

IX. TESTING PROCEDURE RECOMMENDED

In order to secure a test of exact reproducibility, it is desirable that detailed specifications should be observed both as to the form of apparatus and the testing procedure which are to be used. The apparatus recommended for use has been described on page 28 and is illustrated in Fig. 10. No variation from the specifications should be made without very careful consideration, for considerable care has been taken to prepare this apparatus in a form best suited for this work.

It should be noted that the test papers should hang as nearly as possible in the position indicated, namely, midway between the watch glass and the bottom of the upper stopper. If the papers are forced on, merely tearing them in order to put them in position, they will very often be suspended at an angle below the hook; the gas currents through the apparatus are then disturbed and normal results may not be obtained.

For each test the paper should be freshly prepared by submerging it in a 5-per cent solution of lead acetate and then pressing it between two sheets of blotting paper. It should then be hung in the apparatus and the test made at once, the time of exposure being regulated carefully. The gas should flow during test at between 4.5 and 5.5 cubic feet per hour. As soon as the test period is at an end, the flow of gas should be cut off and the test paper at once removed from the apparatus. If no distinct coloration is apparent, the paper should be compared with a similar strip of paper moistened with lead acetate in the same way as described above but not exposed to the gas. Hydrogen sulphide should be reported as present in the gas only when the test paper is *distinctly* darker than the paper not exposed to the gas. In case of uncertainty it is best in official testing work to report hydrogen sulphide as "absent." In other words, lack of conclusive evidence is sufficient to justify a negative report.

In making the test care should of course be taken to use gas fresh from the supply main and to insure that the gas before being tested does not come in contact with water, oil, or other substance by which the small amounts of hydrogen sulphide likely to be present in a commercial gas supply would be removed.

X. COMMERCIAL SIGNIFICANCE OF THE TEST PROPOSED

At the present time very different procedures are followed in testing for hydrogen sulphide and it is, therefore, impossible to say just how the tests which have been studied in the course of the present investigation compare with commercial testing methods in the majority of places. It is certain, however, that the standard procedure which is recommended above will give results which are less severe in their limitation upon a company than many of the tests now used; on the other hand, the procedure is slightly more severe than some of the test methods, particularly that known as the Referees method.

The time of exposure recommended (one minute) conforms to practice commonly found in this country and gives results probably of the desired commercial significance. A one-minute test requires very little time and yet is long enough to prevent any appreciable irregularity due to the unavoidable changes in time of exposure of the paper. Of course, if a more sensitive test is desired, a longer period can be used, or for a less severe test, a shorter period. If any change is made in the testing procedure in order to change the sensitiveness of the test, it is recommended that the period of the test be changed to accomplish this. In any case, the decision as to whether or not it would be desirable to make the test more or less stringent should be considered from an economic standpoint.

The object of the test should be to detect such amounts of hydrogen sulphide as are not commercially permissible, but it is difficult to determine just what limit should be chosen. It is certain that the testing method recommended is such that ordinarily careful operation of any common system of purification will give a gas sufficiently pure to meet the test. Therefore, in practically every case the test can be met without increasing the care now

exercised in the removal of hydrogen sulphide. Occasionally, of course, gas which will not pass the test must be sent out, but only the avoidable irregularities would cause any prolonged difficulty in meeting a test such as is proposed. It is assumed that the regulations under which the testing will be done will be drafted to care for the irregularities which can not be avoided, as suggested in Circular No. 32 of this Bureau. Many tests of similar severity, and some a great deal more severe, have been met regularly in the past, showing that the proposal is certainly not unreasonable.

XI. SUMMARY

The effect of the following variations in the method for the detection of hydrogen sulphide in gas with lead acetate paper has been investigated: (1) The kind of paper, its surface and opacity, method of preparation, strength of lead acetate solution used, and the moisture content of the paper when used; (2) variations due to changes in humidity of the gas, rate of gas flow, and time of exposure to gas; and (3) effect of the form and the size of the apparatus, which determine the gas currents in contact with the paper. A new apparatus and method are described for the continuous preparation of a uniform gas mixture containing a known amount of hydrogen sulphide. The results obtained show both the absolute and the relative sensitivity of the various methods which may be used in official or works laboratories; the quantitative as well as the commercial significance of the results is pointed out and comparisons are made with the results of previous observers. A method is recommended for use which is quick and convenient and gives reproducible results; and a simple and inexpensive form of apparatus is described which can easily be constructed by inexperienced persons without any sacrifice of the advantages due to exactness of specifications. The importance of conforming with the specifications, both as to the apparatus and the procedure in testing, is pointed out.

WASHINGTON, August 19, 1914.

(Continued from second page of cover.)